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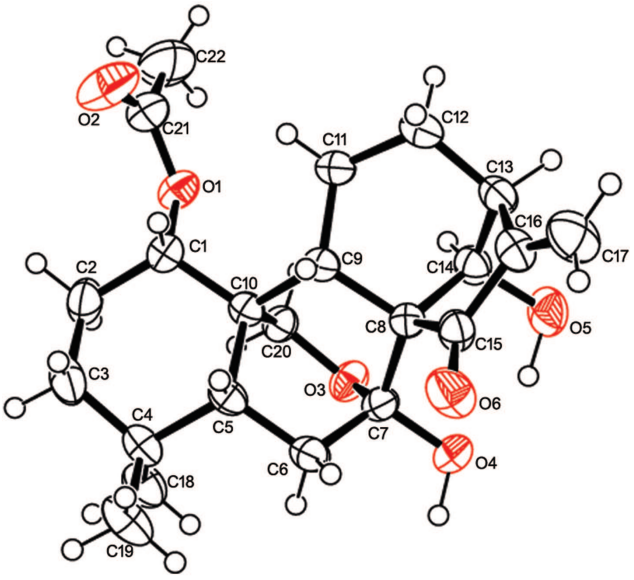
Crystal structure of *ent*-1β-acetoxy-7α,14α-dihydroxy-7β,20-epoxykaur-16-en-15-one, $C_{22}H_{30}O_6$



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Abstract

$C_{22}H_{30}O_6$, monoclinic, $P2_1/c$ (no. 14), $a = 11.1339(16)$ Å, $b = 6.1262(9)$ Å, $c = 14.771(2)$ Å, $\beta = 103.567(5)°$, $V = 1867.9(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0430$, $wR_{ref}(F^2) = 0.1165$, $T = 293$ K.

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The title molecule is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The leaves of *Isodon coetsa* were collected in Kaiyang city of Guizhou province, China in May 2013 and identified by H. J. Zhao at Guiyang College of Traditional Chinese Medicine. The dried and powdered leaves (6 kg) were extracted with MeOH (3x) at room temperature. The residue (800 g) was

Table 1: Data collection and handling.

Crystal:	colorless, block, size 0.21×0.23×0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	CCD, φ and ω scan
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10394, 3287
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2940
$N(param)_{refined}$:	279
Programs:	SHELX [1]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2a	0.681(2)	−0.002(5)	0.866(2)	0.041(6)
H(13)	2a	1.023(2)	0.494(4)	0.664(2)	0.045(7)
H(9)	2a	0.716(2)	0.103(4)	0.709(1)	0.021(5)
H(5)	2a	0.524(2)	0.087(5)	0.701(2)	0.036(6)
H(14)	2a	0.860(2)	0.637(4)	0.746(2)	0.032(6)
H(2A)	2a	0.5138	0.2483	0.9425	0.058
H(2B)	2a	0.5493	0.0041	0.9673	0.058
H(3A)	2a	0.3529	0.0172	0.8688	0.060
H(3B)	2a	0.4425	−0.1046	0.8182	0.060
H(6A)	2a	0.4815	0.3244	0.5845	0.050
H(6B)	2a	0.4178	0.4860	0.6412	0.050
H(11A)	2a	0.8819	0.3528	0.8455	0.049
H(11B)	2a	0.8766	0.0977	0.8374	0.049
H(12A)	2a	0.9737	0.0951	0.7184	0.054
H(12B)	2a	1.0474	0.2700	0.7872	0.054
H(17A)	2a	0.8632	0.1737	0.4553	0.070
H(17B)	2a	1.0017	0.2249	0.5135	0.070
H(18A)	2a	0.3163	0.5085	0.7411	0.076
H(18B)	2a	0.3955	0.4731	0.8427	0.076
H(18C)	2a	0.2627	0.3723	0.8124	0.076
H(19A)	2a	0.3243	−0.0347	0.6608	0.088
H(19B)	2a	0.2714	0.1981	0.6305	0.088
H(19C)	2a	0.2199	0.0638	0.7035	0.088
H(22A)	2a	0.8891	0.3898	1.0750	0.125
H(22B)	2a	0.9913	0.2106	1.0814	0.125
H(22C)	2a	0.8986	0.1975	1.1464	0.125
H(20A)	2a	0.5813	0.5559	0.8615	0.040
H(20B)	2a	0.7219	0.5607	0.8604	0.040
H(4)	2a	0.514(2)	0.722(6)	0.561(2)	0.061
H(5A)	2a	0.762(2)	0.783(6)	0.598(2)	0.062

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Table 3: Fractional coordinates and atomic displacement parameters (Å²).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*a*	0.6493(2)	0.1376(4)	0.8805(2)	0.039(1)	0.033(1)	0.032(1)	0.001(1)	0.0082(9)	0.0018(9)
C(2)	2*a*	0.5335(2)	0.1103(5)	0.9172(2)	0.054(1)	0.050(2)	0.045(1)	0.001(1)	0.022(1)	0.013(1)
C(3)	2*a*	0.4238(2)	0.0359(5)	0.8419(2)	0.046(1)	0.045(2)	0.065(2)	−0.009(1)	0.025(1)	0.004(1)
C(4)	2*a*	0.3909(2)	0.1992(4)	0.7608(2)	0.0308(9)	0.042(1)	0.049(1)	−0.003(1)	0.0150(9)	−0.005(1)
C(5)	2*a*	0.5093(2)	0.2335(4)	0.7236(1)	0.0268(9)	0.034(1)	0.038(1)	−0.0017(9)	0.0085(8)	−0.008(1)
C(6)	2*a*	0.4918(2)	0.4008(4)	0.6433(2)	0.0275(9)	0.059(2)	0.037(1)	0.001(1)	0.0070(9)	0.004(1)
C(7)	2*a*	0.6027(2)	0.5519(4)	0.6570(1)	0.035(1)	0.041(1)	0.031(1)	0.006(1)	0.0069(9)	0.007(1)
C(8)	2*a*	0.7240(2)	0.4299(4)	0.6628(1)	0.0274(9)	0.037(1)	0.029(1)	−0.0019(9)	0.0049(8)	0.001(1)
C(9)	2*a*	0.7364(2)	0.2468(4)	0.7407(1)	0.0268(9)	0.032(1)	0.033(1)	0.0020(9)	0.0058(8)	−0.001(1)
C(10)	2*a*	0.6330(2)	0.2880(4)	0.7952(1)	0.0287(9)	0.031(1)	0.032(1)	−0.0001(8)	0.0071(8)	−0.0016(9)
C(11)	2*a*	0.8684(2)	0.2321(5)	0.8018(2)	0.0304(9)	0.051(2)	0.038(1)	0.005(1)	0.0051(9)	0.008(1)
C(12)	2*a*	0.9681(2)	0.2377(5)	0.7457(2)	0.0276(9)	0.055(2)	0.050(1)	0.003(1)	0.0067(9)	0.003(1)
C(13)	2*a*	0.9393(2)	0.4115(5)	0.6672(2)	0.0284(9)	0.050(2)	0.044(1)	−0.005(1)	0.0100(9)	0.001(1)
C(14)	2*a*	0.8421(2)	0.5748(4)	0.6837(2)	0.034(1)	0.038(1)	0.038(1)	−0.0081(9)	0.0072(9)	−0.002(1)
C(15)	2*a*	0.7380(2)	0.3206(4)	0.5729(2)	0.037(1)	0.038(1)	0.035(1)	−0.006(1)	0.0102(9)	0.000(1)
C(16)	2*a*	0.8730(2)	0.3098(4)	0.5761(2)	0.037(1)	0.039(1)	0.048(1)	−0.004(1)	0.014(1)	−0.001(1)
C(17)	2*a*	0.9169(2)	0.2282(6)	0.5084(2)	0.052(1)	0.070(2)	0.058(2)	0.006(1)	0.023(1)	−0.014(2)
C(18)	2*a*	0.3362(2)	0.4078(5)	0.7922(2)	0.040(1)	0.055(2)	0.062(2)	0.004(1)	0.023(1)	−0.007(1)
C(19)	2*a*	0.2924(2)	0.0970(6)	0.6814(2)	0.034(1)	0.070(2)	0.073(2)	−0.015(1)	0.015(1)	−0.018(2)
C(21)	2*a*	0.8220(2)	0.1062(6)	1.0142(2)	0.047(1)	0.061(2)	0.044(1)	0.006(1)	0.006(1)	0.015(1)
C(22)	2*a*	0.9078(3)	0.2376(8)	1.0856(2)	0.072(2)	0.100(3)	0.062(2)	−0.006(2)	−0.016(2)	0.012(2)
C(20)	2*a*	0.6397(2)	0.5281(4)	0.8234(1)	0.037(1)	0.034(1)	0.030(1)	−0.0002(9)	0.0077(9)	0.0000(9)
O(1)	2*a*	0.7428(1)	0.2354(3)	0.9558(1)	0.0498(8)	0.043(1)	0.0309(8)	0.0019(8)	0.0026(6)	0.0041(8)
O(2)	2*a*	0.8217(2)	−0.0885(5)	1.0084(2)	0.093(2)	0.060(2)	0.074(1)	0.019(1)	−0.005(1)	0.018(1)
O(3)	2*a*	0.6123(1)	0.6688(3)	0.7433(1)	0.0511(9)	0.0311(9)	0.0386(9)	0.0082(7)	0.0124(7)	0.0051(7)
O(4)	2*a*	0.5888(1)	0.7132(3)	0.5882(1)	0.0406(8)	0.060(1)	0.049(1)	0.0086(9)	0.0073(7)	0.024(1)
O(5)	2*a*	0.8360(2)	0.7470(3)	0.6188(1)	0.0498(9)	0.038(1)	0.070(1)	−0.0066(9)	0.0183(9)	0.0097(9)
O(6)	2*a*	0.6567(1)	0.2489(4)	0.5104(1)	0.0452(8)	0.078(1)	0.0416(9)	−0.015(1)	0.0085(7)	−0.017(1)

dissolved in MeOH and ethyl acetate. The crude product was detected by TLC (silica gel), then devided into A-F fractions by silca gel column with dichloromethane/ ethyl acetate (volume ratio 8:2). The title compound was isolated from fraction B over a silica gel column with dichloromethane/acetone (volume ratio 7:3), then the product was dissolved in methanol [2]. After one week transparent crystals were obtained.

Experimental details

Carbon-bound hydrogen atoms were positioned geometrically and refined using a riding model, with d (C—H) = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The hydrogen atoms of the OH groups were refined freely.

Discussion

Many of the *ent*-kaurene diterpenoids isolated from the Isodon plants taste bitter and exhibit various biological activities such as antitumour, antimicrobial and insect growth inhibitory activities [3]. The bond distances and bond angles in the title structure are all in normal ranges. The olefinic bond, ketonic bond and two dihydroxy bonds were clearly identified by the distances d(C16—C17) = 1.311(3) Å, d(C15—O6) = 1.212(3) Å, d(C7—O4) = 1.402(3) Å and d(C14—O5) = 1.421(3) Å, respectively. The oxo-bridge was confirmed by the distances d(C20—O3) = 1.436(3) Å, d(C7—O3) = 1.443(3) Å. The intermolecular connection was achieved through hydrogen bonding with O(4)—H(4)···O6′ angle of 176° and O(5)—H(5A)···O(4) angle of 147°; O(4)···O6′ distance of 2.789(2) Å and O(5)···O(4) distance of 2.691(2) Å (′ = 1−x, 0.5+y, 1−z).

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